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# BIOCERAMICS BASED ON ALUMINUM OXIDE

**V. V. Lashneva,[1] Yu. N. Kryuchkov,[1] and S. V. Sokhan'[1]**

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The composition of $Al_2O_3$-based bioceramic and the technology of bioceramic production are developed. Dental and orthopedic bioceramic implants have passed engineering and clinical tests and are considered serviceable.

The use of ceramic materials substantially expands the possibilities of contemporary medicine in treating a wide range of diseases, including orthopedic, dental, and maxillofacial diseases.

With respect to interaction with the live organism, medical ceramics can be divided into two main categories: bioinert materials which have a minimum extent of chemical, electrochemical, surface-catalytic, and other types of interaction with the organism, and bioactive materials which actively participate in biochemical processes in the organism [1].

The most common among the bioinert materials are ceramics based on aluminum oxide. They are characterized by high biocompatibility and chemical inertness, nontoxicity, high mechanical strength, hardness, and wear resistance. Moreover, they can stay for a long time in a live organism and retain their physical and chemical properties, which creates good prerequisites for their reaction-less implantation and long-term service and makes them highly promising as dental and bone implants.

The achievements of the last years in the field of ceramic technology and sintering theory [4] and the progress in the powder technology [2 – 4] make it possible to perceptibly improve the mechanical properties of ceramics based on aluminum oxide and simultaneously increase its chemical inertness, reliability, and durability.

One of the methods for improving the strength of high-purity grade aluminum oxide is a decrease in the grain size, which is accomplished by the use of single-fraction highly disperse powders with particle size below 1 μm and near-spherical particle shape, as well as by introduction of modifying additives. According to the data in [2], the introduction of a combined additive of MgO and $ZrO_2$, which in sum does not exceed 1%, best contributes to the compaction and formation of fine-grained structure in aluminum oxide ceramics. The mechanism of the effect of these additives on the properties of the alumina-based ceramics is probably determined by the formation of spinel $Al_2O_3 \cdot MgO$ which is located along the grain boundaries impeding the grain growth, as well as by the formation of solid solutions with apperance of vacancies in the anion and cation sublattices and the emergence of substantial stresses in the $Al_2O_3$ lattice due to the difference between the ionic radii of Zr (0.082 nm) and Al (0.059 nm), which intensifies the diffusion and the sintering process.

The present paper describes the results of the development of bioceramics based on $Al_2O_3$ with additives of MgO (0.5 wt.%) and $ZrO_2$ (0.4%) involving the machine treatment of articles after sintering. The initial reactants were $Al(NO_3)_3 \cdot 9H_2O$, aqueous solution of $NH_4OH$, $Mg(NO_3)_2$, $ZrO_2$ (all chemically pure grade), and distilled water.

A highly disperse $Al_2O_3$ powder was obtained by calcination at a temperature of 1250°C of aluminum hydroxide precipitated by the reaction of an aqueous solution of aluminum nitrate with an aqueous solution of ammonia, and subsequent protracted grinding. The aqueous solution of magnesium nitrate was introduced into the aluminum hydroxide, and $ZrO_2$ was introduced into the $Al_2O_3$ during its grinding after calcination. The powder pulverizing was performed by wet grinding for 30 h in a ball mill with a ceramic drum and zirconium dioxide milling bodies. This method of introduction of additives provided for their uniform distribution over the initial powder mixture.

The average particle size in the synthesized powder was 0.3 – 0.4 μm; the particles were nonisometric; the specific area of the powder was 14.0 $m^2/g$ (the BET method). The granulometric composition of the powder determined on a PRO 7000 laser sedimentograph was as follows: content of the fraction of 0.00 – 0.050 μm — 72.87%, 0.50 – 1.00 μm — 13.66%, 1.00 – 2.00 μm — 9.88%, 2.00 – 3.00 μm — 1.59%, > 3 μm — 0.00%.

[1] Institute of Materials Technology Problems, National Academy of Sciences of Ukraine, Kiev, Ukraine; Institute of Superhard Materials, National Academy of Sciences of Ukraine, Kiev, Ukraine.

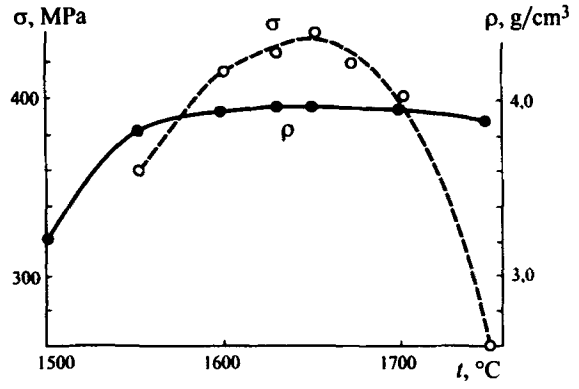


**Fig. 1.** Dependence of density ρ and bending strength σ of bioceramics on sintering temperature *t*.

Samples shaped as rods of 8 × 8 × 50 mm were prepared by semidry uniaxial two-sided compression at a pressure of 100 MPa. The molded samples were fired in air at a temperature of 1250°C for 2 h with a slowly increasing temperature up to the prescribed value, next cooled together with the furnace to room temperature, and sintered in a vacuum of 0.0133 Pa at a temperature from 1500 to 1750°C for 1 – 2 h. The rate of heating and cooling was 300°C/h. After sintering, the sample density, the average grain size, and the bending strength were determined, which made it possible to determine the optimum sintering temperature for obtaining articles with high-quality parameters.

The density of the samples was determined by hydrostatic weighing in kerosene, and the microstructural analysis was performed by electron microscopy. The bending strength was measured using a three-point loading scheme on the TIRAtest machine (with base 35 mm) at a loading rate of 180 mm/min. The sample surface subjected to tensile deformation was polished to grade 12 roughness, and two facets of 0.5 × 45° were applied.

According to the data of chemical, spectral, and x-ray fluorescent analysis, the content of $Al_2O_3$ in the ceramic was over 99.0%; MgO — 0.5%; $ZrO_2$ — 0.4%; $SiO_2$, $Na_2O$, and $K_2O$ in sum not more than 0.1%.

The dependences of density and bending strength versus the sintering temperature obtained in the experiments are shown in Fig. 1. The microstructure (fractograms) of the samples sintered at different temperatures is shown in Fig. 2. The grain size increases from 1 – 2 μm at a temperature of 1550°C to 2 – 3 μm at 1650°C. With a further increase in sintering temperature, the structural homogeneity decreases (at 1720°C, along with grains 2 – 3 μm, some grains up to 7 μm are encountered; at 1750°C against the background of 5 – 6 μm grains, some grains up to 12 – 15 μm are found).

It is seen in Figs. 1 and 2 that the optimum properties are registered in the material sintered at a temperature of 1650°C: σ = 435 MPa; ρ = 3.96 g/cm³; grain size 2 – 3 μm. These parameters satisfy the requirements of ISO 6474 international standard imposed on the alumina-based ceramics used for ceramic implants in surgery.

Owing to the high requirements for precision and surface quality imposed on certain bioceramic products, their technology includes machining after sintering, which calls for the use of special diamond tools due to the high strength and hardness of this material.

A technology for diamond-abrasive microfinishing treatment of semispherical articles up to 32 mm in diameter using the free lapping method has been developed [5, 6]. In the implementation of this method, it is difficult to determine precisely the time needed to bring the article to the required dimensions, since the rate of removal of allowance *l* is unequal for different sites of the article. It is determined by a complex mutual effect of such factors as the contact pressure distribution, the relative motion speed, etc., and also depends on the selected technological parameters: cutting velocity, clamping force *Q*, angular rotation velocity $\omega_1$ of tool *1*, angle β between *Q* and angular rotation velocity $\omega_2$ of tool *2*, and

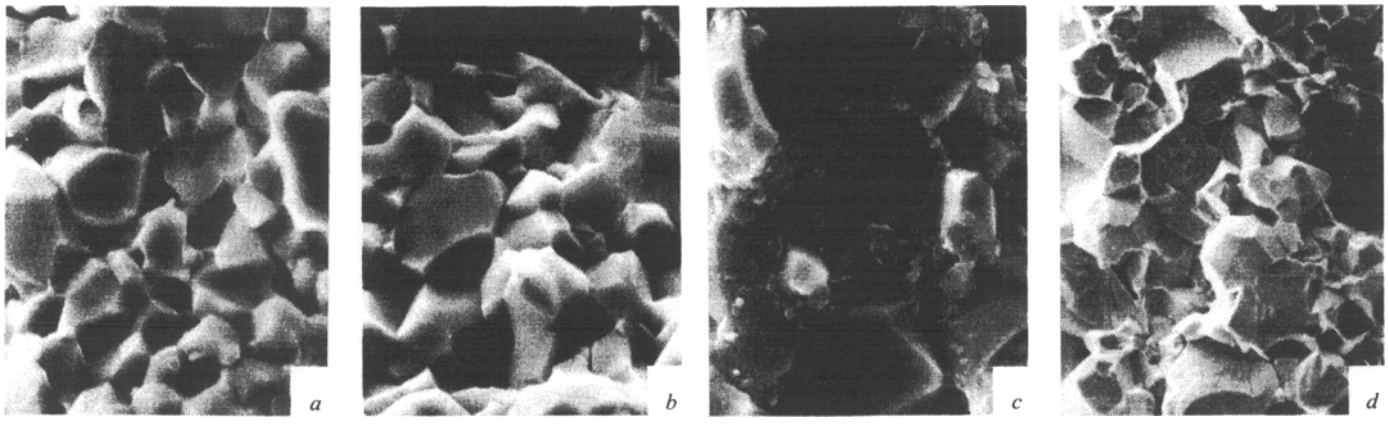


**Fig. 2.** Fractograms of samples sintered at different temperatures: *a*) sintering temperature 1550°C, sintering duration 2 h (×6000); *b*) 1650°C, 1 h (×6000); *c*) 1720°C, 1 h (×6000); *d*) 1750°C, 2 h (×1000).

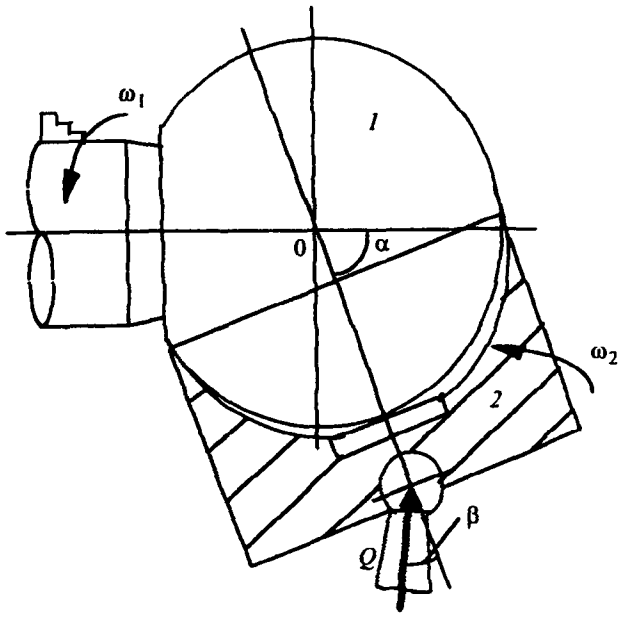


Fig. 3. Scheme of microfinishing treatment process.

change in the area of the contact between the tool and the article.

The scheme of the microfinishing treatment process is given in Fig. 3. The difficulties related to the determination of the moment when the article is within the limits of permissible deviation were overcome by adjusting the angle β.

The controlling parameters of the process varied within the following limits: clamping force $Q$ — from 10 to 100 N, angle α from 0 to 90°, angle β from 0 to 14°.

The performed investigation revealed that the rate of removal of the allowance increases in the axial direction $I_a$, and decreases in the radial discretion $I_r$, with an increase in the angle β.

The results obtained for a rotational speed of the tool of 1250 $min^{-1}$ and a clamping force of 80 N are shown in Fig. 4. It can be seen that with $\beta < 9°$, the allowance removal in the radial direction is predominant ($I_r > I_a$), with $\beta > 9°$, the allowance removal in the axial direction dominates, and when β is approximately 9°, the equidistant removal of allowance from the entire surface of the product is observed.

Thus, it is possible to control the processing of the article by adjusting the angle β, i.e., first, the optimum value for the angle between the vectors of the clamping force and the angular rotational speed of the tool is determined depending on the allowance distribution over the surface of the article, and then the duration of the treatment is calculated.

In the course of the experiment, ceramic implants were made which are used in stomatology as supports for fixed dentures (dental implants) and in traumatology and orthopedies as a capitulum of hip joint prosthesis (orthopedic implants).

The stomatological implants have a cylindrical, conical, or laminar shape and a developed surface, which allows for

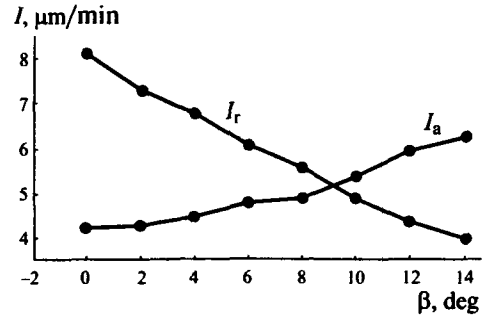


Fig. 4. Effect of variation in the angle β on the rate of allowance removal $I$ in the radial $I_r$ and axial $I_a$ directions.

reliable fixation in the bone. Their standard sizes provide for anthropometric variations found in human jaws and do not require machine treatment in implantation. In particular, the height of the cylindrical dental implants varies from 10 to 15 mm and their diameter is 5 mm, and the height of the laminar implants varies from 15 to 20 mm, their width from 16 to 22 mm, and the thickness is 1.8 mm. The weight of the implants is about 1 g.

The capitula of hip joint prosthesis have a semispherical shape 28 and 32 mm in diameter (negative tolerance for the diameter is 0.02 mm, deviation from the spherical shape < 5 μm), and the height of the capitula is 25 and 29 mm, respectively, with the internal conical opening up to 21 mm deep, for the capitulum to be set on the prosthesis stem. The roughness of the capitulum spherical surface Ra is not more than 0.16 μm. The weight of the implants is 25 and 55 g, respectively.

The dental and orthopedic implants (the latter as part of hip joint prosthesis) were subjected to all the required technical and clinical tests (registration certificate 370/97).

Thus, the performed investigations made it possible to obtain bioceramics based on aluminum oxide that are distinguished by high physicomechanical parameters: $Al_2O_3$ content > 99.0%, density of 3.96 $g/m^3$, grain size < 2 – 3 μm, bending strength 435 MPa.

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